Near Infra-Red Based Online Mineral Phase Analysis of Bauxite

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Abstract

Near Infra-red technology is used by laboratory scale instruments for mineral phase analysis of bauxite ore, to distinguish (for example) between Kaolinite, Gibbsite, Quartz and Boehmite as sources of Alumina and Silica, or to distinguish between reactive and non-reactive Silica. The representivity of laboratory analyses of large-scale flows or inventories of bauxite depends highly on representative sampling, and the availability of these laboratory analyses often involve long delays. Online analyzers are commonly used in mining applications to get an early indication of the actual mined ore quality. Most available technologies can only analyze elemental (often presented as oxide equivalent) concentrations. Near Infra-Red (NIR) based online analysis overcomes that limitation and gives an online mineral phase analysis. Through its mineralogical analysis, the technology can provide accurate analysis of alumina available and the reactive silica along with other mineral composition. It does so without using any radioactive or gamma radiation sources, making the technology safe and without equipment operating limitations. For the user, the direct and online analysis of the bauxite's different mineral phases gives significant advantages, such as using the composition of the mine product for developing and validating block-models for the mine. Specific tests of mine areas by feeding the crusher with the target material and monitoring the analyzer results, allow monitoring of reactive silica content on a stockpile and to react accordingly. It allows to use more variable raw materials for building a stockpile, while still allowing a satisfactory understanding of the instantaneous feed quality and to react with appropriate process control settings in the refinery. This paper and presentation will showcase impressions and results from the first installation in a bauxite mine, where it is presently in operation.

Keywords: Online analysis, reactive silica, mineral phase analysis, near infra-red, bauxite, alumina.

1. Introduction

Shareholder value and reduction of total costs of ownership and production are key drivers in today's world. Despite a recovery in the LME Aluminum price, the demand and supply situation continue to put pressure on most mining companies to work as efficiently as possible. This includes utilization of raw materials from the mine and elsewhere, and of production process additives, their logistics and associated maintenance processes.

Today there is much talk about Industry 4.0, which in practice comes down to increased automation, improved communication and monitoring, along with self-diagnosis and new levels of analysis [1]. These technologies enable faster decision-making in a closed-loop control, resulting in smoother and more efficient processes, requiring less and less human intervention.

Online analyzers are a basic tool for the move towards Industry 4.0, and higher automation levels. Looking at today's mines, trucks and their drivers are being replaced by automated equipment to produce equivalent results. This demands different monitoring further downstream to evaluate if the mined material is useful for further processing, how it shall be utilized, mixed or even rejected. Conventional ‘offline’ methods like sampling and laboratory analysis are often too slow for that decision-making process to keep the operation cost efficient.

2. Online Analysis of Bauxite

Bauxite ore is usually mined from multiple areas simultaneously. These areas may have different mineralogy and consequently different available alumina and reactive silica contents. To enable a stable feed to the refinery or bauxite shipment, these raw materials from different sources need to be blended. Usually the only information to ensure a proper blend in real time is the drill hole analysis for the corresponding zone, so an online analysis of the mined bauxite ensures a better blend and avoids exceptional peaks.

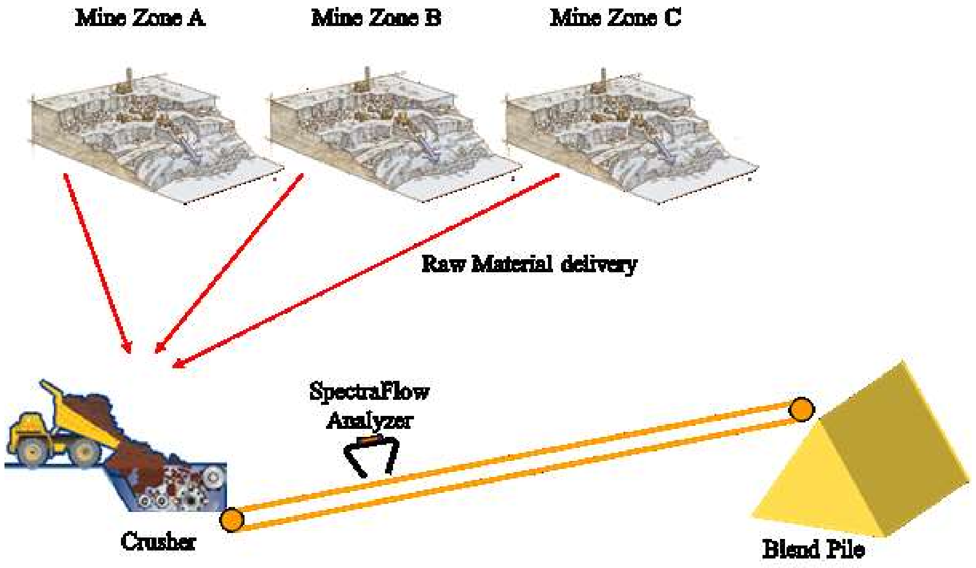


Figure 1. General setup of an online analyzer for pile blending.

The same setup can be the case in a refinery where different bauxite types are processed and where ore types may be blended to create an optimized (quality and cost) refinery feed material.

Online Analyzers are typically characterized by the type of energy source they are using, such as a radioactive source, neutron tube, laser, or a near infra-red (NIR) source presented here. A comparison of the technologies will follow in section 2.5

2.1 Near Infrared Technology for Online Analysis

A still relatively new technology in the market for the online analysis of minerals is based on a near infra-red (NIR) source: a simple halogen light bulb. NIR is electromagnetic radiation with a wavelength range of 700 nm to 2500 nm, possibly emitted by a light bulb or just plain sun light.

The Near Infra-Red (NIR) source gives the technology its name. In NIR applications, light emitted from the halogen bulb shines on the material for analysis. The material absorbs part of the light, more precisely part of the wavelength spectra emitted by the NIR source. Each molecule of the material being analysed absorbs the light at its specific and characteristic wavelength. What is not absorbed is reflected, and this reflection is detected by a lens of an FTIR spectrometer. This spectrometer analyses the absorption (by difference) and returns the corresponding absorbed spectra.

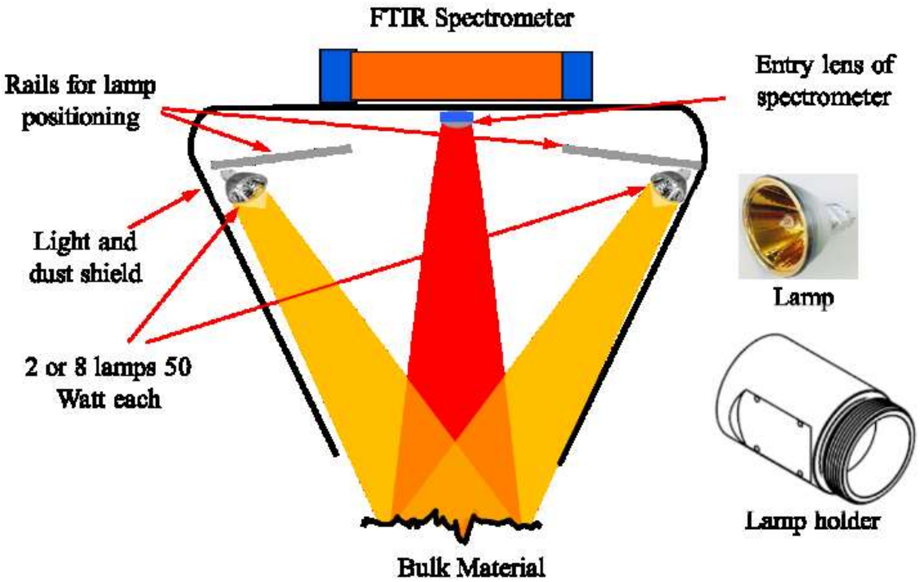


Figure 2. Schematic of the measurement process of the NIR online analyzer technology.

An important aspect of the NIR technology is that it is a surface analysis. The light in the NIR application does not pass through or even penetrate the material beyond the surface layer. The consequence is that for an accurate analysis;

a) material needs to be statistically homogeneous on the belt being analyzed; this is the case as the material goes through a crusher and falls (from a statistical point of view) in random order on the belt.
b) a very high measurement frequency is required to ensure that all material is measured; the analyzers takes a measurement every 428 milli-seconds (ms) and averages these results over one minute.

Under these conditions, and specifically avoiding installation in locations where material classification may occur, the SpectraFlow Crossbelt analyzer has been proven to work very accurately in cement and iron ore applications. Typically, the online analyzer is located immediately after a crusher, or for example, immediately before a stockpile. Figure 3 shows a truck bringing material with larger pieces of ore with different mineralogy. In the crusher these materials are crushed and mixed, opening many rock faces. These are measured afterwards by the NIR analyzer. This is of course, a simplification for the purposes of demonstrating the value of crushing the feed ore to promote greater homogeneity and exposure of the mineral phases present.

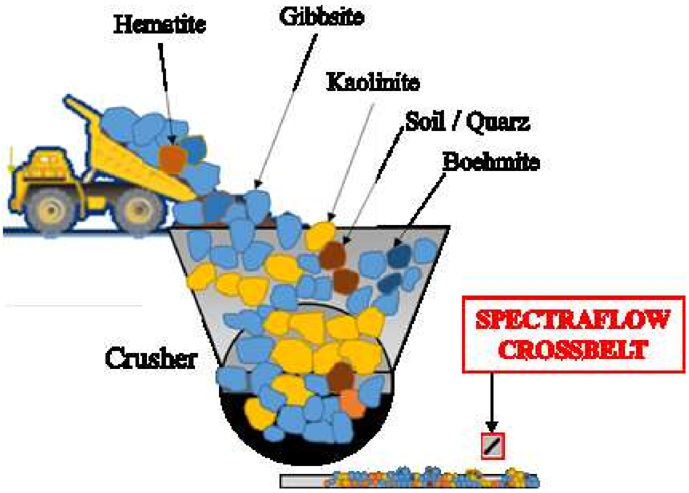


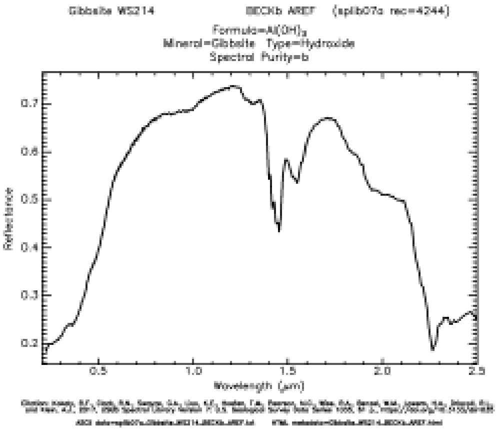
Figure 3. The homogenization of raw material inside the crusher.

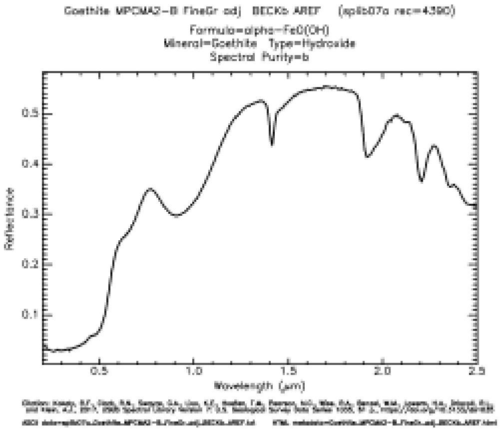
The NIR analyzer scans the surface of the presented material every 428 ms, equaling 140 scans per minute, and averages the results over one minute periods. At an average belt speed of 4 m/s, this equals 240 meters of raw material on the belt, or at a crusher throughput of 1500 t/h, approximately 25t of raw material per minute. This high frequency gives the SpectraFlow system very robust and representative measurements.

2.2. NIR as a Mineral Phase Analysis

The main difference between NIR and other online technologies is that near infra-red is sensitive to mineral phase composition, while most other online analyzers measure elemental composition.

The energy in the near-infrared range is absorbed on the belt according to the material's molecular composition including minerals, any organics, and moisture. Every mineral has its own unique signature in the NIR spectra as illustrated in the graphs below:





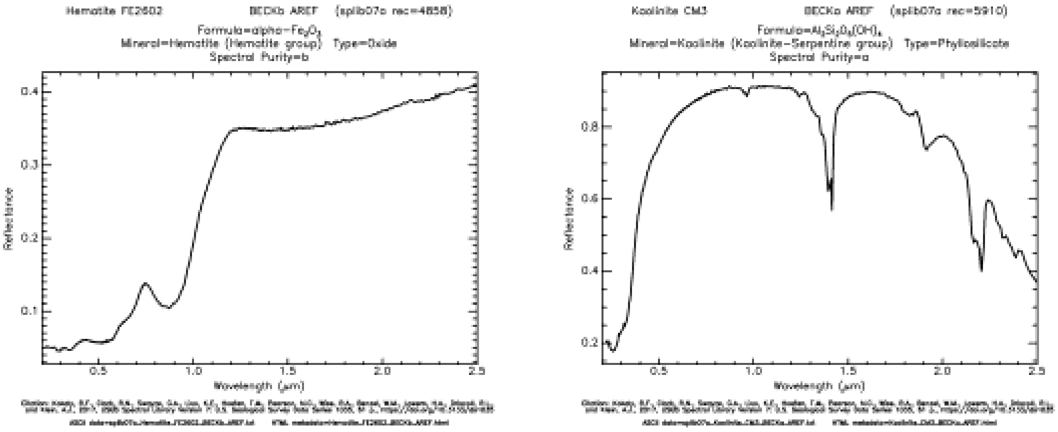


Figure 4. Examples of NIR Spectra for Gibbsite, Goethite, Hematite and Kaolinite [2].

Determining mineralogical composition by NIR Spectroscopy is the basis for SpectraFlow technology. In terms of mineralogical sensitivity, a wide range of constituents may be analyzed or interpreted: including available alumina, reactive silica or the mineral phases directly. To put this analytical capability in an online analyzer that is robust enough to provide a 24/7 operation in different climates and work environments was a challenge that SpectraFlow have successfully solved.

2.3 Calibration of NIR

Usual calibration routines for other online analyzers often involve some kind of artificial standards, but this is not the approach and does not work well with NIR technology. Calibration in online NIR projects is rather based on actual material compositions found at the customer's site. One of the first steps in a project is to collect samples from the different mine areas, as well as the mixes on the conveyor belt. In this way, individual materials as well as blends of these are available for the calibration. The goal of the calibration procedure is to 'merge' the chemical / mineralogical composition of the samples with the spectral information.

Two factors need to be considered during this calibration procedure:

1) The material's moisture is also influential in the calibration, and with changing moisture levels, the wet chemical / mineralogical composition is also changing. So, if moisture levels of the materials on the conveyor belt are changing, it is not enough to take the spectral information at a given moisture level. The spectral information must be collected at multiple moisture levels for one and the same sample and the provided chemical / mineralogical composition (on a dry basis).
2) NIR is a surface analysis and that needs to be accounted for during the calibration procedure. To do that, the samples are mixed and measured multiple times at the same moisture level while the sample holder is moving. This way the dynamics of the process as well as different presentations of the samples can be covered.

For the calibration procedure, SpectraFlow requires samples at their laboratory in Switzerland. The samples are dried and then measured four times at each relevant moisture level, starting with 0 % (dry). The samples are put on a tray which is rotating underneath the analyzer. The one-minute measurement is performed to collect the 140 spectra which are combined to give the spectra for the minute. The sample is taken aside, mixed and measured again – four times and then repeated at the next moisture level.

With the spectral information and the chemical / mineralogical results of the samples available, the calibration is performed by mathematical algorithms. The calibration models are validated with a test set and once approved, loaded into the analyzer in the plant.

Figure 5. Calibration setup: NIR detector and sources.

2.4 Long Term Stability and Influencing Factors for a Near Infra-Red Online System

On a day to day basis, the stability and resulting accuracy of the NIR online system is influenced by the source, detector, material presentation, temperature and humidity.

A first step after loading the calibration to the newly installed analyzer is to define offsets, to exactly account for a large portion of the difference between the laboratory and real operating situation. During this period, the analysis of the online analyzer is compared with actual values retrieved for example by a sampler or by a back-calculation of a stockpile. That way material presentation, possible segregation, crushed size, etc., is accounted for.

As mentioned, humidity also influences the NIR analytics, and varying material moisture levels are accounted for in the calibration. SpectraFlow compensates in the calibration model.

The temperature of the material is usually stable or with little variation. Air temperatures may change tremendously, from day to night or throughout the seasons. Outside temperature does not affect the calibration, but it may affect the sensor. For that reason, SpectraFlow ensures the sensor is temperature controlled.

Source and detector also play an important role in the long terms stability. In the SpectraFlow setup, the sources are exchanged once they deplete to around 95 % of their original energy level, which is typically after nine to fifteen months, depending on the accumulated operating hours. During this period, every two months a referencing procedure is performed to account for the aging effect.

On the detector side, the spectrometer used by SpectraFlow is highly stable and allows a calibration model developed on the laboratory equipment to be transferred without adjustments to the analyzer in the field.

2.5 Comparison of Online Analyzer Technologies

Online Analyzers are available from a range of suppliers, but usually based on the same technologies. Below is a comparison of the key aspects of the different technologies that are available for online analysis [3]:

Table 1. Comparison of online analysis technologies.

	PGNAA / CNA	**XRF**	**LIBS**	**NIR**
Analysis Basis	Nucleus	Electron Shells	Nucleus	Molecules, Mineral Phases
Reports	Oxides	Oxides	Oxides	Mineral Phases or Oxides
Sources	Radioactive (Cf_{252}) or Neutron Tube (with Deuterium and Tritium)	X-Ray Tube	Laser	Halogen light bulbs
Detectors	2, typically sodium iodide (NaI) crystals with photo-multiplier tube NaI-Scintilation counter	1, silicium drift detector	1 or more, Spectrometer, depending on analytical range	1, IR Spectrometer
Elements Possible	From Na (Cf source) or from O (neutron tube),	From Na for small particle size < 30 mm, from K for larger particle size	All	All elements incl. H, moisture
Additional analyzers required	Yes – Moisture	Yes – Moisture and possibly for distance	Yes – moisture	No
Average particle size	< 150 mm	< 25 mm	< 150 mm	<150 mm
Recalibration required	With change of source	Regular basis	Regular basis	Only with change in Mineralogy
Type	Transmission	Surface, mm spot	Surface, mm spot	Surface, covering the conveyor belt width

Dependency	Belt speed, belt load (volumetric)	Constant distance between detector and surface required, representative and smooth surface, possible in bypass systems	Homogeneity of the material on the surface	No nonlinear layering
Accuracy on specific constituents	Low on MgO and Na2O	Depends on material presentation and particle size	Poor on elements with low periodic number, Sulphur (S) not possible	High on all oxides / minerals
Import / Operating Licence	Required, complicated and forbidden in some countries	Required, simple	Required, simple	Not needed
Weight / Static requirements	High, >1200 kgs, plus shielding	Low ~ 50 kgs	Medium, ~ 400 kgs	Low, ~150 kgs
Operating cost	PGNAA: High due to cost for sources and calibrations CNA: Medium for sources but high on electrical	Medium	Medium	Low

In summary, the near infra-red technology is the only one able to return mineralogical information, in the case of bauxite, to distinguish between different silica or alumina sources. It has a low operating cost, and a stable analyzer.

3. Application of NIR Online Analysis on Bauxite Ore

3.1 University Study

SpectraFlow Analytics (at the time still part of ABB) performed a first detailed feasibility study in 2012 together with the Institute of Mineralogy and Petrography of the University of Innsbruck.

The scope of the study was to verify that bauxite NIR-spectra contain all the mineralogy information for determining qualitative and quantitative composition. The influence of the crush size on the prediction results was also evaluated.

To cover the granularity differences of the material and to see if coarser particles provide enough representative surface, samples received from a bauxite mine were sorted in 3 size fractions: below 5 cm, from 5 to 10 cm and above 10 cm.

For the qualitative analysis, the samples (total of 198 lateritic bauxite samples from Brazil) were analyzed by X-ray powder diffraction (XRPD) and refined by a quantitative phase analysis using Rietveld. The result showed varying levels of kaolinite, gibbsite, goethite, hematite and anatase. In a next step the samples as well as pure minerals were analyzed by a bench top infrared spectrometer.

The investigation has shown that near infra-red spectroscopy is sensitive enough to capture all the mineralogical information needed to determine the content of minerals in bauxite. A calibration model was established randomly dividing the 198 samples into a calibration and validation set.

The combined prediction results of Calibration and Validation sets of kaolinite and gibbsite, which the study finally focused on, are presented with their validation sets below in Figure 6.

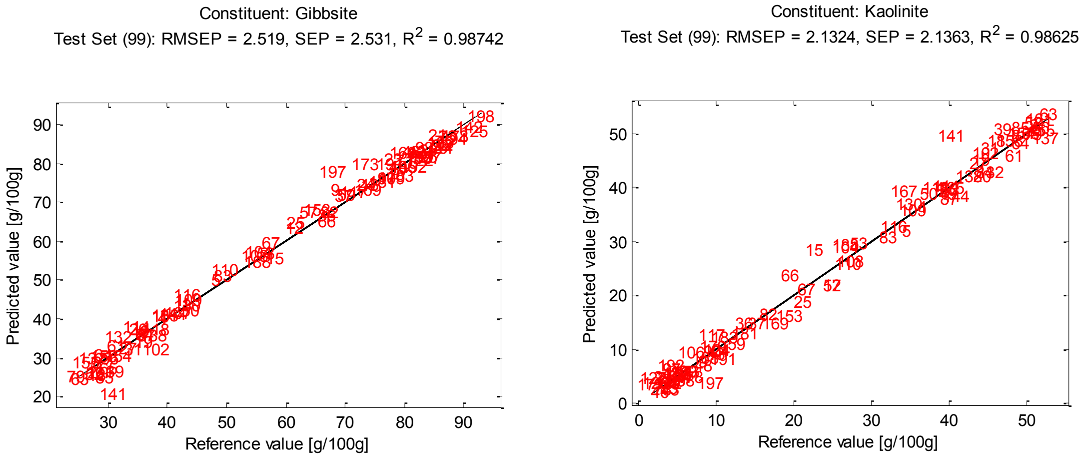


Figure 6. Results of the validation set for gibbsite and kaolinite as part of the study.

The test set shows different parameters for evaluation:

- RMSEP: root mean squared error of prediction
- SEP: standard error of prediction
- R^2: coefficient of determination – an indicator to show the quality of the calibration model, ideally the value should be "1"

RMSEP or SEP is the simplest and most efficient measure of the uncertainty in NIR predictions. This value is a measure of the average uncertainty that can be expected when predicting new samples. RMSEP is expressed in the same units as the predicted parameter. The results of future predictions can then be presented as predicted values ± 2 x RMSEP. This measure is valid provided that the new samples are similar to the ones used for the calibration otherwise the prediction error might be much higher [4].

Other lateritic bauxites originating from Australian, Jamaican, Surinam and Arkansas were predicted based on the established model above. It is not a procedure we would do today as there are still mineralogical differences, however the actual vs. prediction results for Gibbsite and Kaolinite can be seen below:

Table 2. Prediction results of four other lateritic bauxites based on the Brazilian model.

	Gibbsite			**Kaolinite**		
	Reference	Predicted	Delta	Reference	Predicted	Delta
Australian	56.54	56.24	0.30	18.81	15.14	3.67
Jamaican	72.85	74.87	-2.02	1.48	5.82	-4.34
Surinam	78.47	78.52	0.05	8.14	6.22	1.92
Arkansas	57.23	55.01	2.22	28.85	27.03	1.82

Karstic bauxites of European origin were also evaluated as part of the study. Despite OH-bands of Boehmite and Diaspore plus the small discrete OH-bands of Kaolinite, being hardly observable in the NIR spectra, the prediction of the Kaolinite (for example) showed good results.

These fundamental investigations based on standard laboratory equipment clearly show that near infrared spectroscopy is suitable for the quantitative determination of the major components in bauxite raw materials (i.e. gibbsite and kaolinite) with sufficient precision and accuracy.

3.2 Feasibility Studies

Further feasibility studies were done by SpectraFlow using their industrial spectrometer. The results of two studies are outlined below:

In 2017 there was a follow up evaluation to the University based study above. Six samples were received from the same mine in 2017 (5 years after the original study). The samples have a granularity ranging from <50 mm to <0.37 mm. An analysis of three blind samples was provided.

The calibration model which was obtained from the laboratory NIR equipment (with the very wide calibration ranges) was now used to predict the 6 samples for Gibbsite and Kaolinite:

Table 3. Prediction results of known and blind samples.

Sample	Remarks	Granulometric Range	**Gibbsite**		**Kaolinite**	
			Actual as provided by client	Prediction by SFA	Actual as provided by client	Prediction by SFA
Bauxite I	Known Sample	<50.0 >12.7mm	55.15	54.4	2.44	1.9
Bauxite II	Known Sample	<0.37mm	10.16	13.7	27.3	30.7
Bauxite IIII	Known Sample	<12.7mm	36.96	34.2	11.69	11.33
Bauxite IV	Blind Sample	<50.0 >12.7mm	46.14	45.5	6.18	4.6
Bauxite V	Blind Sample	<12.7mm >0.84mm	53.07	55.4	4.38	2.1
Bauxite VI	Blind Sample	<12.7mm	35.47	35.7	10.64	6.7

The actual values on the blind samples were provided by the client after the prediction. Considering that there was no prior calibration on that type of spectrometer, the long timeframe between calibration, the new samples and the wide calibration range (Gibbsite 20 – 90 %, Kaolinite 0 – 50 %), the prediction results are considered very good, and the show that there is no loss in analytical quality due to the granulometry.

In a second feasibility study on bauxite done in 2017, SpectraFlow received 13 known and 3 blind samples of a lateritic bauxite with the goal to predict the available alumina and reactive silica concentration. The twelve raw material samples were split in two and with the known sample, there were a total of 25 samples for the calibration and validation procedure. The following plots (Figures 7 and 8) show the calibration curves on basis of 19 training samples:

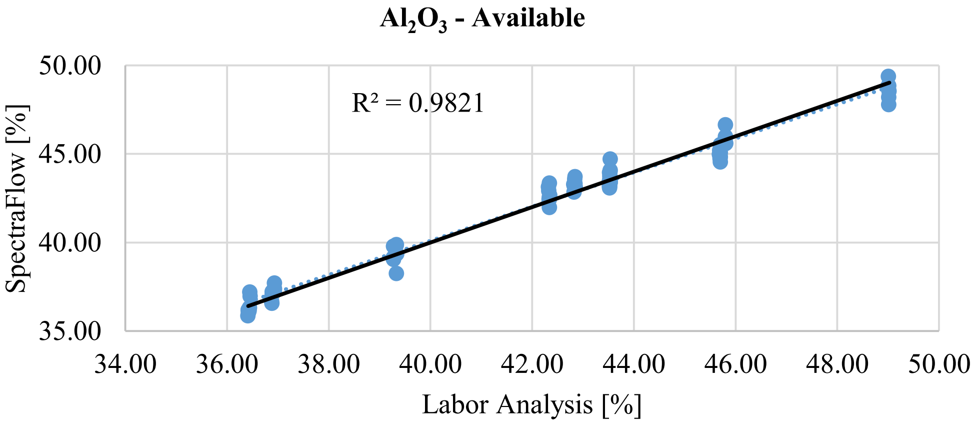


Figure 7. Available Al_2O_3 calibration curve of the training samples.

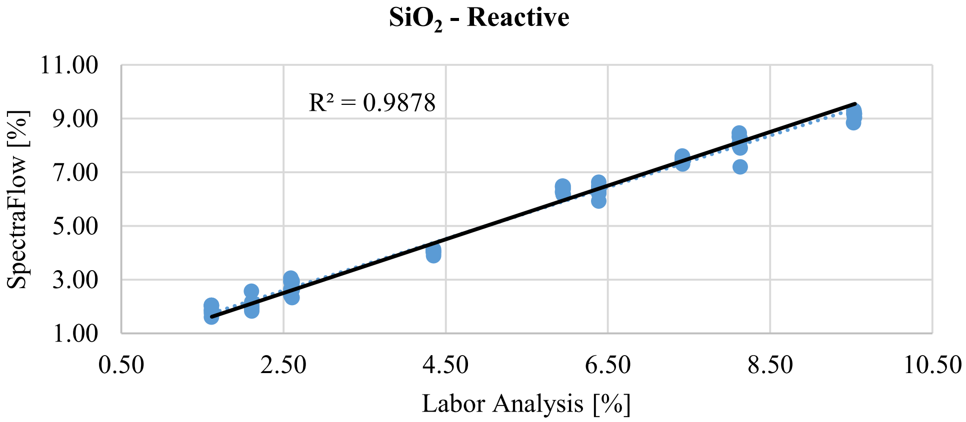


Figure 8. Reactive silica calibration curve of the training samples.

The result of blind sample prediction is shown below in Table 4. Although the calibration sample set was very limited, the correlation between the reference results and predicted SpectraFlow measurements are high.

Table 4. Results of blind sample prediction.

Sample		Contents (%)			
		Al_2O_3-Av	SFA prediction	SiO_2-Re	SFA prediction
BM	Bauxite Mix - Blind	**48.99**	**48.6**	**2.32**	**2.2**

14	Sample				
BM 15	Bauxite Mix - Blind Sample	**38.95**	**35.4**	**7.14**	**7.6**
BM 16	Bauxite Mix - Blind Sample	**46.34**	**47.5**	**1.55**	**1.5**

3.3 Current Project (Under Execution)

SpectraFlow is currently executing an installation in a mine / refinery in Australia. To date, the analyzer is not yet installed, but the calibration work based on 140 raw material and mix samples is done.

At this site, the lateritic bauxite consists of a low-silica greenstone and high silica granite-derived material. The major minerals are gibbsite, goethite, hematite, quartz and kaolinite. Minor components are anatase, corundum, boehmite and maghemite. Impurities include oxalate (and other organic carbon compounds), sulfates and fluoride.
Mining is conducted from multiple pits that are within hauling distance of the crusher. The grade varies between each area, so the balancing of the major grade parameters like available alumina, reactive silica, total carbon, sulfate and oxalate from the simultaneously mined areas is a constant challenge [5].

With the experience of FTIR analytical equipment in the laboratory, the plant decided to install a SpectraFlow NIR online analyzer to get a more consistent feed grade. Multiple installation points were discussed, but for this first installation a location in the refinery was selected. This location also features an existing sampler and will consequently allow a better evaluation of the accuracy of the online analyzer.

As the installation location has parallel conveyor belts, part of the project will be for the plant to install a frame so that the online analyzer can be moved from one to the other belt for analysis. As there is no shielding requirement, this can be done easily

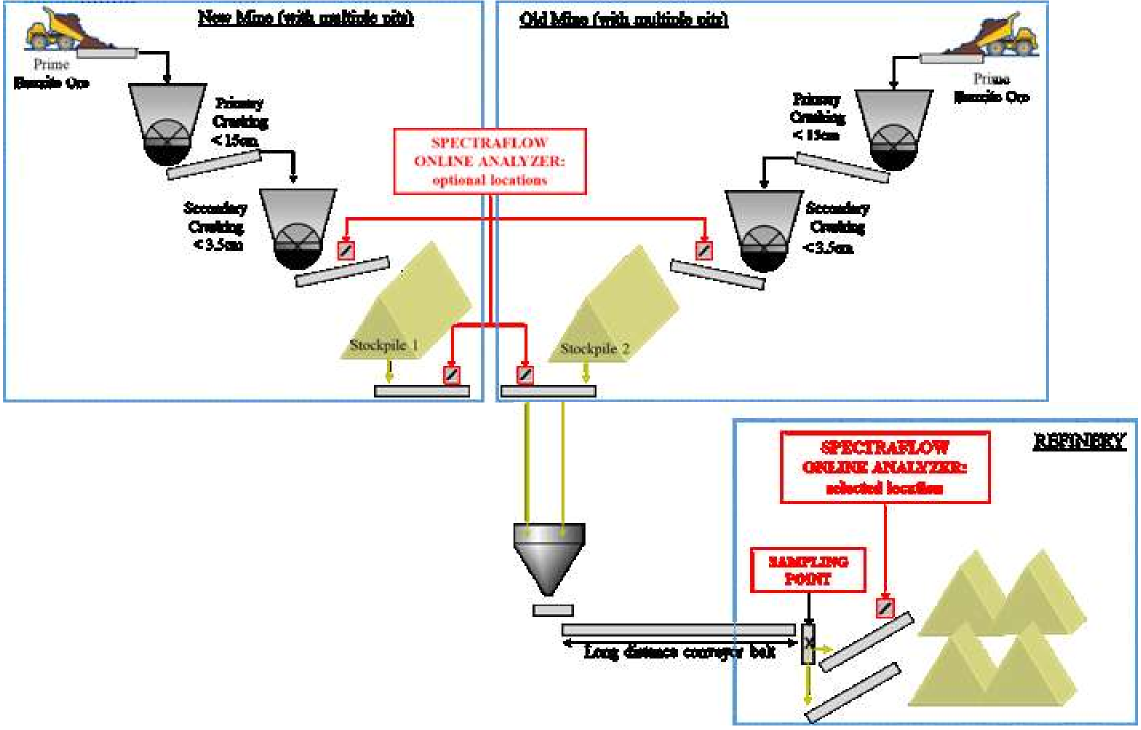


Figure 9. Installation location schematic of the SpectraFlow NIR online analyzer.

Figure 10. Installation location.

The samples received covered the calibration range are noted in Table 5 below.

Table 5. Excerpt of the constituents and their calibration range.

%	Available Alumina	Al_2O_3	Reactive Silica	SiO_2	Sodium Oxalate	Total Carbon
Min	2.2	20.83	0.2	0.69	0.01	0.11
Max	43.4	49.37	26.9	34.9	0.43	0.76

The NIR spectral information of the samples was retrieved by the calibration procedure described in section 2.3. The training set consisted of 1550 data points. The test set consisted of 700 data points. The calibration results can be seen below in Figures 11 to 16.

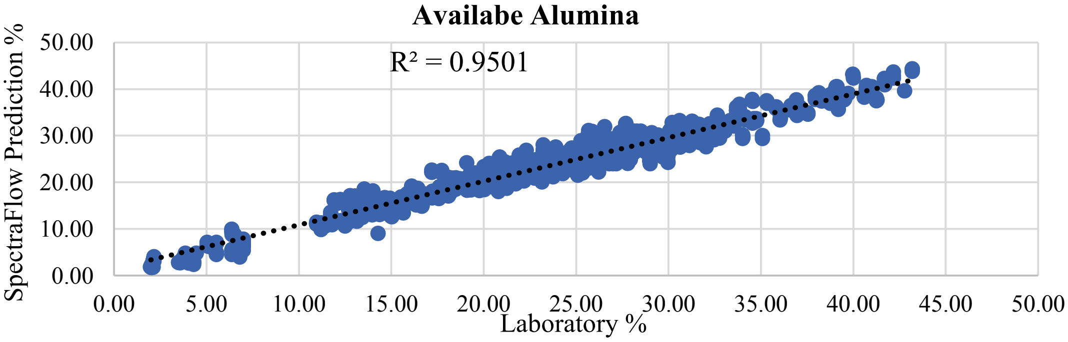


Figure 11. Calibration result for available alumina.

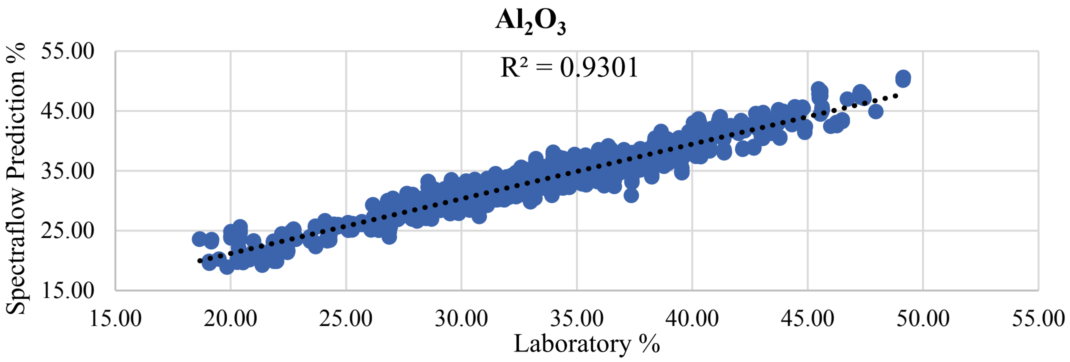


Figure 12. Calibration result for total alumina.

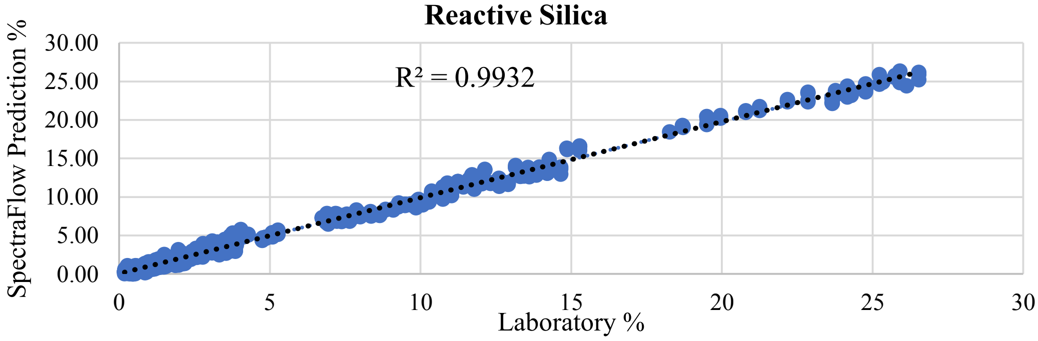


Figure 13. Calibration result for reactive silica.

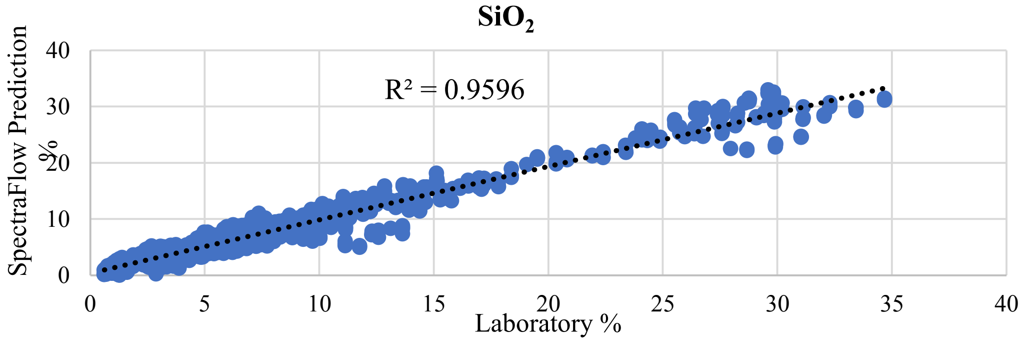


Figure 14. Calibration result for total SiO_2.

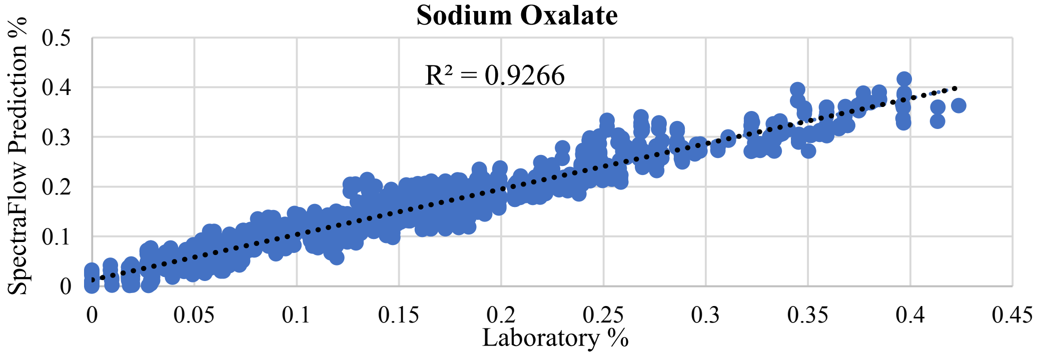


Figure 15. Calibration result for sodium oxalate.

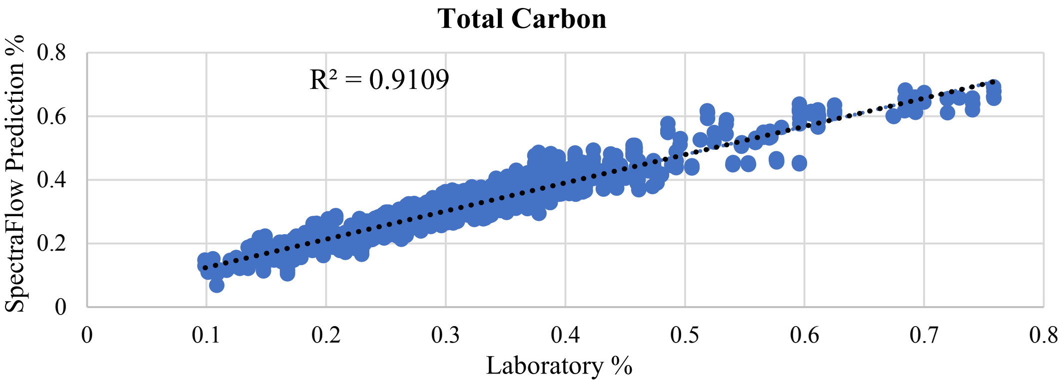


Figure 16. Calibration result for total carbon.

Table 6 below serves as an overview of the results of the validation set.

Table 6. Overview of the calibration results.

%	Available Alumina	Al_2O_3	Reactive Silica	SiO_2	Sodium Oxalate	Total Carbon
Calibration Range						
Min	2.2	20.83	0.2	0.69	0.01	0.11
Max	43.4	49.37	26.9	34.9	0.43	0.76
Training Set						
R^2	0.95	0.93	0.99	0.95	0.92	0.91
SEP	1.63	1.43	0.4	1.36	0.02	0.03
Validation Set						
R^2	0.95	0.93	0.99	0.96	0.92	0.92
SEP	1.67	1.45	0.4	1.37	0.02	0.03
RMSEP	1.67	1.45	0.4	1.37	0.02	0.03
SEP in % of calibration range	4.05 %	5.08 %	1.50 %	4.01 %	4.76 %	4.62 %

The following interpretation can be made from the numbers in Table 6:

- The SEP in training and validation set is almost identical. This means that the validation samples fit the training set.
- The RMSEP and the SEP in the validation samples is identical. This indicates that there is no slope in the calibration.
- R^2 is equal high and equal on all constituents. This shows that the calibration model built on the samples is very robust.

With an expected Gaussian distribution, the SEP above equals one sigma.

Due to operation limitations, the installation of the analyzer is scheduled for late 2018. Results of the operation will be reported in later publications.

4. Conclusion

The above work shows that the SpectraFlow NIR Online Analyzer can be used for online bauxite analysis for ore blending to reduce variations in the feed to the refinery or to reject certain raw materials and for refinery process control.

The NIR technology, known from laboratory instruments using the same principle, can distinguish between different mineral phases and can analyze organic as well as inorganic material in a direct way without using back-calculations.

The radiation free analyzer with halogen light bulbs as sources and a spectrometer as detector allows operation without permits or specialized radiation officers on site. The design can be light since it requires no shielding and allows installation everywhere without the need for structural reinforcements and makes the analyzer easily movable to alternate between conveyor belts.

The online availability is proven in installations in the cement and iron ore industry, which have similar temperature and dust conditions to bauxite mines and alumina refinery bauxite stockpile areas.

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